

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS
AND INTERFERENCES

Ex parte MARK ALAN ROSENZWEIG
and
JEFFREY ALLAN PFAENDTNER

Appeal 2008-1020
Application 10/707,465¹
Technology Center 1700

Decided: 17 March 2008

Before ADRIENE LEPIANE HANLON, THOMAS A. WALTZ, and
CAROL A. SPIEGEL, *Administrative Patent Judges*.

SPIEGEL, *Administrative Patent Judge*.

DECISION ON APPEAL

¹ Application 10/707,465 ("the 465 application") was filed 16 December 2003. The real party-in-interest is said to be the General Electric Company ("GE") (APPEAL BRIEF UNDER 37 CFR §41.37, filed 18 January 2007 ("Br."), 3).

I. Statement of the Case

Mark Alan Rosenzweig and Jeffrey Allan Pfaendtner ("Appellants") appeal under 35 U.S.C. § 134 from the Examiner's final rejection of claims 1-20, all of the pending claims. We have jurisdiction under 35 U.S.C. § 6(b). We AFFIRM.

The subject matter on appeal relates to a process for removing "adherent particles," formed *in situ* during aluminizing, from an aluminized surface with an aqueous caustic hydroxide solution, such as a potassium hydroxide (KOH) solution. Claim 1 (Br. 31) is illustrative and reads:

A process comprising the steps of:

forming an aluminized surface within an internal cavity of a component by placing within the internal cavity a material comprising metallic particles of an aluminum source and then heating the material and the component, wherein during heating some of the metallic particles oxidize and sinter to form adherent particles that are sintered to the aluminized surface; and then

contacting the aluminized surface with an aqueous caustic hydroxide solution until the adherent particles are removed from the surface.

In addition to the admitted prior art ("APA") in Appellants' specification, the Examiner relies on the following references² as evidence of unpatentability:

Shurman	US 5,707,453	Jan. 13, 1998
Fernihough	US 6,265,022 B1	Jul. 24, 2001
Schilbe	US 6,475,289 B2	Nov. 5, 2002
Chen	US 2005/0035086 A1	Feb. 17, 2005

² No references to *et al.* are made in this opinion.

Appellants rely on their joint declaration under 37 C.F.R. § 1.132 ("Declaration") as evidence of patentability.

Claims 1-10 and 14-20 stand rejected under 35 U.S.C. § 103(a) as obvious over APA in view of either Schilbe or Fernihough (Ans.³ 3-4). Claims 11-13 stand rejected under 35 U.S.C. § 103(a) as obvious over APA in view of either Schilbe or Fernihough, as applied to claim 1, and further in view of Shurman (Ans. 4-5).

Claims 1-11 and 13-20 stand rejected under 35 U.S.C. § 103(a) as obvious over APA in view of Chen (Ans. 5-6). Claim 12 stands rejected under 35 U.S.C. § 103(a) as obvious over APA in view of Chen, as applied to claims 1 and 11 above, and further in view of Shurman (Ans. 6).

Since Appellants have not separately argued the patentability of any of claims 1-20, we decide this appeal on the basis of claim 1. 37 C.F.R. § 41.37(c)(1)(vii).

The issue is whether one of ordinary skill in the art would have reasonably expected the particle removal process of Schilbe, Fernihough and/or Chen to remove the specific adherent particles recited in the claimed process.

II. Findings of Fact ("FF")

The following findings of fact and any set out in the Discussion are supported by a preponderance of the evidence of record.

A. The 465 Specification

1. background of the invention, i.e., the APA

- [1] According to the 465 specification ("Spec."), it is common practice to coat internal surfaces of turbine engine components, e.g., internal

³ Examiner's Answer mailed 31 May 2007 ("Ans.").

cooling passages of a turbine blade, with aluminide coatings to provide oxidation and hot corrosion resistance (Spec., ¶¶ 2 and 4).

- [2] Known aluminizing processes are said to involve (a) making a slurry by mixing (i) a powdered metallic aluminum source, e.g., CrAl, (ii) an activator, e.g., alkali metal halide, and (iii) an inert oxide dispersant, e.g., alumina or zirconia, in a liquid vehicle; (b) injecting the slurry into the internal cavities of the component and drying the liquid vehicle to dry coat the component; and, (c) heating the coated component to at least 1700°F (~930°C) so that the activator vaporizes and reacts with the aluminum source to form a volatile aluminum halide, which then reacts with the component's surface to form an aluminide coating (Spec., ¶ 3).
- [3] Remnants of unreacted, adherent components of the slurry must be removed to maximize air flow through aluminized passages of an air-cooled component (Spec., ¶ 4).
- [4] Specifically, particles of the aluminum source which have oxidized and sintered⁴ to the aluminized surfaces as a result of the high aluminizing temperatures are said to be "very difficult to remove" (Spec., ¶ 4).

⁴ According to the McGraw-Hill CONCISE ENCYCLOPEDIA OF SCIENCE & TECHNOLOGY, 4th ed., McGraw-Hill publishers, New York (1998), pp. 1764, sintering is the "welding together and growth of contact area between two or more initially distinct particles at temperatures below the melting point, but above the one-half of the melting point in kelvins. Since the rate of sintering is greater with smaller than with larger particles, the process is more important with powders, as in powder metallurgy and in firing of ceramic oxides."

- [5] Known approaches for removing unreacted, adherent slurry solids include (a) high-pressure water jet flushing the component and (b) autoclaving (subjecting to high temperature and high pressure) the component with caustic compounds (Spec., ¶ 4).
- [6] The latter approach is said to "have been used with limited success" and the former approach is said to be "undesirable in view of the cost of autoclaving operations" (Spec., ¶ 4).

2. the invention

- [7] The 465 specification describes a method said to be "particularly suited for the removal of oxidized particles that form as a result of oxidation of the aluminum source powder used in slurry aluminiding [sic] processes, wherein the oxidized powder particles become attached by sintering to the aluminized surface" comprising "contacting the aluminized surface with an aqueous caustic hydroxide solution until these adherent particles are removed therefrom" (Spec. ¶ 5).
- [8] In one embodiment, the aluminized surface is (a) contacted with a 100 g/l aqueous potassium hydroxide solution at about 66°C to about 88°C for at least two hours, preferably with ultrasonic agitation, (b) rinsed with water, and (c) dried (Spec., ¶ 10).

B. Schilbe

- [9] Schilbe discloses chemically cleaning "accumulated foreign material including surface oxides and/or dirt on the internal cooling air passage surfaces" of "an engine-run gas turbine engine airfoil" (Schilbe, 2:38-40 and 59-64).

- [10] Schilbe's cleaning method comprises immersing the airfoil in an inorganic hydroxide medium at superambient temperature and atmospheric pressure for no more than 360 minutes to remove the accumulated oxides and dirt without chemically damaging the airfoil (Schilbe 1:49-63).
- [11] One embodiment of Schilbe's cleaning method uses an aqueous solution of 50% (w/w) potassium hydroxide at a minimum temperature of at least about 275°F (135°C) (Schilbe 3:37-42).

C. Fernihough

- [12] Fernihough discloses plugging cooling holes of a gas turbine component with a ceramic slurry or paste with essentially no polymerizing agent included; heating the entire assembly to a temperature that partially sinters the ceramic material; depositing a coating, such as MCrAlY, on the external surface of the turbine component; removing the ceramic plug from the cooling holes, e.g., by heating the components in a vacuum furnace at 600°C for 2 hours; and, removing residual ceramic particles by selective chemical cleaning, e.g., with potassium hydroxide (Fernihough, 2:58 through 3:27; 5:35-6:30).

D. Shurman

Shurman describes using focused ultrasonic energy to assist in cleaning internal cavities of gas turbine engine airfoils (Shurman 2:52-64; 4:38-43).

E. Chen

- [13] Chen describes upgrading an aluminide coating on a used turbine engine component to a platinum aluminide coating by (i) cleaning at

least one surface of the component to remove hot corrosion products without damaging the aluminide coating, (ii) depositing a layer of platinum on the cleaned surface, and (iii) forming a second aluminide coating on the surface of the component (Chen, ¶¶ 7-15).

- [14] According to Chen, its method removes hot corrosion products without damaging or removing the underlying aluminide coating (Chen, ¶ 23).
- [15] In some embodiments, the cleaning step comprises contacting degraded aluminized surfaces with aqueous caustics, e.g., about 10% to about 50% potassium hydroxide solutions, at a temperature of about 60°C to about 100°C for a time ranging from about 20 minutes to about 4 hours (Chen ¶¶ 37-39).

F. The Examiner's position

- [16] The Examiner found that the APA differed from the process of claim 1 in failing to teach contacting the aluminized surface with an aqueous caustic hydroxide solution until the adherent particles are removed from the surface (Ans., 3 and 5).
- [17] The Examiner concluded that, in view of Schilbe, it would have been obvious for one of ordinary skill in the art "to use the caustics (KOH) well known in the art for removal of strongly adherent oxides from the internal surfaces of turbine components without damaging superalloy materials" (Ans., 3).
- [18] The Examiner also concluded that, in view of Fernihough, it would have been obvious to one of ordinary skill in the art "to use (KOH) after an aluminizing process in order to suitably remove strongly

adhering residual metal oxides (ceramic) from the internal surfaces of turbine components" (Ans., 3).

- [19] The Examiner concluded too that, in view of Chen, it would have been obvious "to use the caustic solution of Chen et al. since Chen et al. teaches that the solution is well suited for removal of the strongly adherent alumina scale and other metal oxides that form on the internal surfaces of turbine components without damaging underlying coatings" (Ans., 5-6).

G. Appellants' position

- [20] Appellants argue that the dirt deposits, hot corrosion products, and ceramic plugs removed by Schible, Chen, and Fernihough, respectively, are not the same as the adherent sintered oxide particles removed by the claimed process (Br., 19 and 26).
- [21] According to Appellants, the processes of Schible, Chen, and Fernihough "do not provide a reasonable expectation of success for using a caustic hydroxide solution to remove oxide particles that are not just sintered to a surface, but are metallic particles that are oxidized, sintered, and aluminized to a surface" (Br., 19 and 26).
- [22] Appellants rely on their Rule 132 Declaration to provide a technical explanation of the differences between the particles removed by their claimed process and the particles removed by Schible, Chen, and Fernihough (Br., 19 and 26).

H. Appellants' Rule 132 Declaration

- [23] According to Appellants,

While the use of KOH and other aqueous caustic hydroxide solutions to remove engine-deposited dirt (such as Schilbe and Shurman), hot corrosion

products (such as Chen), and ceramic plugs (such as Fernihough) may have been known, the contaminants removed by Shilbe, Shurman, Chen, and Fernihough are so different from the oxidized, sintered, and aluminized particles we were trying to remove that it was not obvious to us or our co-inventors that a caustic solution such as KOH would work (Declaration, ¶ 8).

- [24] According to Appellants, the particles they are trying to remove are "firmly attached" to the aluminized surfaces because they "form and adhere in-situ through a combined process of oxidation of the particles, sintering of the particles, and aluminization of the walls" (Declaration, ¶ 7).
- [25] Citing internal confidential GE reports said to document the nature of dirt deposits in aircraft engines, Appellants testified that

(a) The nature of dirt deposits (such as Schilbe and Shurman) is significantly different from aluminum oxide formed in-situ during aluminizing. . . . Oxide particles from engine dirt and debris typically comprise components based upon aluminosilicates modified with iron, magnesium, calcium, potassium, and other potential impurities. While pure alumina may be present in the dirt deposits, many other chemicals are also present. The microstructure of these dirt deposits may range from polycrystalline to amorphous or even glassy. The interface with the base metal is typically well defined with only limited interaction with the deposit, since the operating environment on the internal cavities is less than about 1600°F. [Declaration, ¶ 8(a), emphasis added.]

- [26] Citing other internal confidential GE reports said to document the nature of hot corrosion deposits, Appellants testified that

(b) The nature of hot corrosion deposits (such as Chen) **is** also significantly different from aluminum oxide formed in-situ during aluminizing. . . . Hot corrosion products are formed by definition in the presence of corrodants, typically a sodium and sulfur containing compound. The nature of hot corrosion is to break down and flux the protective alumina scale that forms on bare or coated nickel-based superalloys. The scale formed typically is a mixture of alumina plus nickel and chromium oxides. The scale may contain impurities contributed by the corrodant, (primarily Na and S), from the base metal (Ti, Ta, W, Mo, etc.), and from the ever-present engine dirt (Ca, Fe, Mg, K, etc.). The microstructure of hot corrosion products is typically voluminous, layered, porous, and non-adherent, which contribute to the rapid degradation from hot corrosion. While the corrosion products are formed by chemical reaction with the base metal or coating, the interface temperatures typically are less than 1700°F at the corrosion front.
[Declaration, ¶ 8(b), emphasis added.]

- [27] Appellants still further testified that "[t]he nature of the ceramic plug materials as described in Fernihough **is not entirely clear**. . . .

[However,] the plug material of Fernihough **can be inferred to be** porous, not well bonded to the substrate, formed at low temperatures, and possibly containing many constituents other than pure alumina" (Declaration, ¶ 8(c), emphasis added).

- [28] Appellants opine that the material removed according to the claimed process is pure alumina which bonded well to the surface of the

turbine component because of the high heat of in-situ formation (1700°F and greater) and by the nature of the growth of atomic diffusion (Declaration, ¶ 8(d)).

III. Discussion

A. Issue and Legal Standard

An invention is patentable unless "the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains." 35 U.S.C. § 103(a). A determination of obviousness considers both whether a person of ordinary skill in the art would have had reasonably combined the prior art to achieve the claimed invention and whether there would have been a reasonable expectation of success in doing so. *Brown & Williamson Tobacco Corp. v. Philip Morris, Inc.*, 229 F.3d 1120, 1124 (Fed. Cir. 2000).

Here, it appears to be undisputed that aqueous caustic hydroxide, e.g., potassium hydroxide, solutions were known to remove accumulated turbine engine dirt (Schilbe), hot corrosion products (Chen), and ceramic plugs (Fernihough). The dispute is whether there would have been a reasonable expectation that a process using aqueous caustic hydroxide solutions to remove the materials described by Schilbe, Chen, and Fernihough from an aluminized surface would also remove "adherent particles," formed *in situ* as a result of the aluminizing process (APA).

The Examiner deems the materials described by Schilbe, Chen, and Fernihough to be strongly adherent metal oxides "firmly attached through a combined process of oxidation of the particles, sintering of the particles at high temperature and bonding with an aluminide surface" (Ans., 8).

According to the Examiner, "[t]he oxide particles of Schilbe, Chen and Fernihough references are also at high temperature and sinter or bond to the outside of the aluminized surface" (Ans. 8-9).

Appellants contend that adherent particles formed *in situ* while aluminizing a surface are basically pure alumina (Al_2O_3) (FF 28) which is "not just sintered to a surface, but . . . oxidized, sintered, and aluminized to a surface" (FF 21). In other words, the *in situ* formed adherent particles of the claimed process are "very difficult to remove" (FF 4). Essentially, Appellants' position is that a process which removes particles not as well bonded to an aluminized surface would not reasonably be expected to remove particles more strongly bonded to the aluminized surface. Appellants rely on their Declaration to establish that the materials described by Schilbe, Chen, and Fernihough are not as well bonded to an aluminized surface as *in situ* formed adherent particles.

B. Rejections based on Schilbe

Appellants testified that accumulated engine dirt deposits, such as described by Schilbe, have microstructures ranging from polycrystalline to amorphous or even glassy; and, since the operating environment of the engine's internal cavities is less than about 1600°F, a well defined interface is formed with only limited interaction with base metal (FF 25). Thus, the temperature at which accumulated engine dirt deposits form is less than the at least 1700°F temperature used in the APA for aluminizing a surface (FF 2). We find Appellants' testimony that accumulated engine dirt deposits, such as described by Schilbe, are significantly different from and less well bonded to aluminized surfaces than *in situ* formed adherent particles to be credible. The Examiner has not established otherwise.

Furthermore, the Examiner's reasonable expectation of success of combining the Schilbe process with the APA to achieve the claimed process is based on the same material (aluminum and other metal impurities) being bonded to the same aluminide surface of a turbine component at high temperatures and under similar process conditions (Ans., 8). However, the evidence of record suggests the process conditions are not similar.

Aluminizing process temperatures are at least 1700°F, while accumulated engine dirt deposits form at temperatures less than about 1600°F (FF 2 and 25). Accumulated engine dirt deposits may or may not be particulate and have only limited interaction with the base metal vis-à-vis aluminized particles (FF 25). Thus, the Examiner's reasonable expectation of success is without sufficient factual basis.

Shurman does not overcome the deficiencies in Schilbe. Therefore, based on the foregoing, we REVERSE the rejections of claims 1-10 and 14-20 under § 103(a) as obvious over APA in view of Schilbe and of claims 11-13 under § 103(a) as obvious over APA in view of Schilbe, as applied to claim 1, and further in view of Shurman.

C. Rejections based on Chen

Appellants testified that the microstructure of hot corrosion deposits, such as described by Chen, "is typically voluminous, layered, porous, and non-adherent" (FF 26). We find Appellants' testimony that hot corrosion deposits, such as described by Chen, are significantly different from *in situ* formed adherent aluminized particles and are non-adherent to aluminized surfaces to be credible. The Examiner has not established otherwise. Indeed, the background section of Chen characterizes hot corrosion products as condensing on components' surfaces (Chen, ¶ 3).

Again, the Examiner's reasonable expectation of success of combining the Chen process with the APA to achieve the claimed process is based on the same material (aluminum and other metal impurities) being bonded to the same aluminide surface of a turbine component at high temperatures and under similar process conditions (Ans., 8). However, the evidence of record indicates that the hot corrosion products of Chen are not bonded to aluminized surfaces of turbine engine components. Thus, the Examiner's reasonable expectation of success is without sufficient factual basis.

Shurman does not overcome the deficiencies in Chen. Therefore, based on the foregoing, we REVERSE the rejections of claims 1-11 and 13-20 under § 103(a) as obvious over APA in view of Chen and of claim 12 under § 103(a) as obvious over APA in view of Chen, as applied to claims 1 and 11 above, and further in view of Shurman.

D. Rejections based on Fernihough

Appellants testified that the nature of the ceramic plug material in Fernihough is "not entirely clear" and opine that the ceramic plug material "can be inferred to be porous, not well bonded to the substrate, formed at low temperatures, and possibly containing many constituents other than pure alumina" (FF 27). However, Fernihough discloses plugging cooling holes of a gas turbine component with a ceramic slurry or paste; heating the entire assembly to a temperature that partially sinters the ceramic material; and, depositing a coating, such as MCrAlY, on the external surface of the turbine component (FF 12). Only after the coating deposition, e.g., MCrAlY, is the ceramic plug removed from the turbine component's cooling holes (FF 12). Then "residual" ceramic particles are removed from the cooling holes by selective cleaning with potassium hydroxide (FF 12). According to

Fernihough, the plug material may advantageously "withstand the high temperatures of the diffusion heat treatment necessary for the bonding between base material of the component and the MCrAlY coating" (Fernihough, 3:18-25). Thus, as noted by the Examiner, Fernihough appears to teach heating the plug material, performing an aluminizing process and removing only the residual ceramic with potassium hydroxide (Ans., 7). Appellants proffer no evidence of the nature of the interface between the Fernihough's plug material and the base metal of the turbine component after the MCrAlY coating step or other indicia of the plug product formed *in situ* by Fernihough's process. Therefore, we accord Appellants' testimony on the nature of Fernihough's plug product formed *in situ* by Fernihough's process little, if any, weight. Facts established by rebuttal evidence must be evaluated along with the facts on which the conclusion of a *prima facie* case of obviousness was reached, not against the conclusion itself. *In re Eli Lilly*, 902 F.2d 943, 945 (Fed. Cir. 1990). On balance we find that the Examiner has established a *prima facie* case of obviousness of the claimed process over the combined disclosures of the APA and Fernihough.

Therefore, based on the foregoing, we AFFIRM the rejections of claims 1-10 and 14-20 under § 103(a) as obvious over APA in view of Fernihough; and, of claims 11-13 under 103(a) as obvious over APA in view of Fernihough, as applied to claim 1, and further in view of Shurman.

ORDER

Upon consideration of the record and for the reasons given, it is

ORDERED that the decision of the Examiner rejecting claims 1-10 and 14-20 under 35 U.S.C. § 103(a) as obvious over APA in view of Schilbe is REVERSED;

FURTHER ORDERED that the decision of the Examiner rejecting claims 11-13 under 35 U.S.C. § 103(a) as obvious over APA in view of Schilbe, as applied to claim 1, and further in view of Shurman is REVERSED;

FURTHER ORDERED that the decision of the Examiner rejecting claims 1-10 and 14-20 under 35 U.S.C. § 103(a) as obvious over APA in view of Fernihough is AFFIRMED;

FURTHER ORDERED that the decision of the Examiner rejecting claims 11-13 under 35 U.S.C. § 103(a) as obvious over APA in view of Fernihough, as applied to claim 1, and further in view of Shurman is AFFIRMED;

FURTHER ORDERED that the decision of the Examiner rejecting claims 1-11 and 13-20 under 35 U.S.C. § 103(a) as obvious over APA in view of Chen is REVERSED;

FURTHER ORDERED that the decision of the Examiner rejecting claim 12 under 35 U.S.C. § 103(a) as obvious over APA in view of Chen, as applied to claims 1 and 11 above, and further in view of Shurman is REVERSED;

FURTHER ORDERED that no time period for taking any subsequent action in connection with this appeal may be extended under 37 C.F.R. § 1.136(a)(2006); and,

FURTHER ORDERED that the case be returned to the Examiner for action consistent herewith.

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No time period for taking any subsequent action in connection with this appeal may be extended under 37 C.F.R. § 1.136(a).

AFFIRMED

Appeal 2008-1020
Application 10/707,465

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